High Temperature Conductivity Measurements in Doped YbCo$_2$Sb$_{12}$ Skutterudites

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4/30/2010

The following experiment examines the change in conductivity of indium-doped Yb-Co-Sb skutterudites from temperatures ranging from room temperature to 500 Kelvins. The concepts behind the conductivity, such as band gaps, intrinsic and extrinsic charge carriers, and molecular structure, are explained. Several other thermoelectric effects are discussed, with the ultimate goal of determining the figures of merit of the materials.
A senior thesis submitted in partial fulfillment of the requirements for the degree of Honors Bachelor of Science in Physics

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2010

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I. Theory and Background

Motivation

As is evident from their name, semiconductors are materials that have and electrical conductivity in between that of metal conductors and insulators, giving them some interesting properties. Whereas charge in conductive metals is only carried by electrons, charge in semiconductors can be carried through combinations of electrons and “holes”, or the absences of electrons. Although semiconductors have been around just as long as the other elements, their inherent usefulness has only just recently been realized with the advent of solar cells, diodes, and computer chips. The huge market and profit potential has lead to both mass production of semiconductors and a flood of research on the subject. For example, the Semiconductor Industry Association, representing 85% of United States semiconductor manufacturers, reports profits of $115 billion in the US alone and $226 billion worldwide, good for a 51% market share. Silicon is the most commonly used material for commercial semiconductors, but other popular choices include germanium and gallium arsenide.

Figure 1. Left: A collection of quantum dots. Right: Silicon computer chips.

Semiconductors are heavily researched in labs across the world. Although the omnipresence of computers today creates substantial demand for semiconductors, there are many other ways to utilize the materials. Among the potential uses are quantum dots, wires, and wells. Quantum dots are created
by layering different semiconductors to produce one-, two-, or three-dimensional electron traps. These structures can in turn be used in quantum computing, high-energy lasers, and transistors, as well as in biotechnology. For example, research at the Berkeley National Laboratory demonstrated a way to synthesize non-toxic quantum dots in order to probe cells and genes. Solar cells are also an active area of research. Using a junction of different types of semiconductors a p-n junction (to be discussed later) can be created to absorb sunlight, which can be converted into electrical charge, as shown in Figure 2. There is an ongoing push to make solar cells increasingly more efficient, less expensive, and lightweight. The Georgia Tech Research Institute has made breakthroughs in the area, producing three dimensional solar cells that allow more harvested photons over a smaller area.

These are only a few examples of the multitude of uses for semiconductors. Semiconductors have become ubiquitous in modern technology, and the demand for them will only continue to increase. It is therefore important to continue the search for newer and better semiconductors. The semiconductors
in this experiment are known as skutterudites, and their properties will be discussed in the experimental section. The next sections will establish the foundations of semiconductor physics, and allow a greater understanding of the results of the experiment.

**Electrical Conductivity**

As stated previously, semiconductors have an electrical resistivity in between that of metals and insulators, in the range of $10^{-2}$ to $10^9$ ohm-cm [Kittel]. The voltage drop across any material given a current $I$ is given by the well-known equation:

$$V = IR \ (1)$$

where $R$ is the resistance of the particular sample. However, this resistance is dependent both on the intrinsic properties and the geometry of the material. The equation

$$R = \rho \frac{l}{A} \ (2)$$

introduces the resistivity of the material, and is independent of the geometry of the material. This equation makes logical sense: a larger $\rho$, a longer sample, or a smaller cross-sectional area will all result in a more resistant sample.

The quantity that will be explored in detail in this lab is the intrinsic resistivity of the material, or more conveniently, its inverse, known as the conductivity:

$$\sigma = \frac{1}{\rho} \ (3)$$

What makes a sample more conductive than another? Take equation (1) above, substituting (2) for $R$
\[ V = l \frac{I}{\sigma A} \quad (4) \]

But \( V/L \) is simply another expression for the electric field \( E \). The current term in the equation can also be replaced by the number and velocity of charge carriers, the charge they are carrying, and the area which they are flowing through:

\[ I = n q v_d A \quad (5) \]

Substituting this in, the areas cancel and left over is

\[ E = \frac{n q v_d}{\sigma} \quad (6) \]

Now the velocity term can be eliminated. Using the impulse-momentum equation, another expression for \( v_d \) can be obtained.

\[ m v = F \Delta t \quad (7) \]

The force is related to the electric field by \( F = q E \) and \( \Delta t \) is simply the mean time between particle collisions, or the drift velocity \( \tau \)

\[ v_d = \frac{q E \tau}{m} \quad (8) \]

Substituting this back into (6), an expression for the conductivity independent of the electric field is produced

\[ \sigma = \frac{n q^2 \tau}{m} \quad (9) \]

Therefore, the conductivity of a material depends linearly on the number of charge carriers and the mean collision time between. But what exactly is meant by a “charge carrier”? As will be seen, there are
two ways in which charge can travel through a semiconductor, intrinsic conduction and extrinsic conduction. Intrinsic conduction refers to the electrical properties of the semiconductor itself, whereas extrinsic conduction is a result of impurities introduced into the sample. Both these topics will be discussed in the next two sections.

Intrinsic Conduction and Band Gaps

Intrinsic conductivity is prominent in highly purified samples, and is highly dependent on the band gap and the temperature of the material. Each chemical element has a certain number of electrons, and these electrons fill up energy bands in a highly specific fashion. The band gap is the difference in energy between the lowest unfilled band (the conduction band) and the highest filled band (the valence band), as shown in the figure. The smaller the band gap, the less energy is required for an electron to jump between bands.

In equation form:
\[ E_g = E_c - E_v \] (10)

Temperature also plays an important role. At 0 K, no charge carriers are able to cross the barrier, as they all inhabit the lowest possible states. Increasing the temperature will impart more thermal energy to the electrons and increase their ability to cross the band.

In order to obtain a simple expression for the density of charge carriers in terms of the band gap and the temperature, a nearly free electron model is assumed, which results in a simple parabolic model for the band edges in terms of the wavevector, as shown in equation (11).

The energy of a conduction band electron in terms of \( k \) is given by

\[ \epsilon = E_c + \frac{\hbar^2 k^2}{2m_c} \] (11)

where \( m_c \) is the reduced mass of an electron in the conduction band.

The chemical potential \( \mu \) is used in the Fermi-Dirac distribution to model the number of electrons excited from the valence band to the conduction band.

The Fermi-Dirac distribution is given by:

\[ F_e(\epsilon) = \frac{1}{1 + e^{(\epsilon - \mu)/kT}} \] (12)

Assuming that \( \epsilon - \mu \gg kT \), the above expression reduces to:

\[ F_e(\epsilon) \approx e^{(\mu - \epsilon)/kT} \] (13)

The density of electron states in three dimensions, as given by Kittel, are

\[ D_e(\epsilon) = \frac{1}{2\pi^2 \left( \frac{2m_c}{\hbar^2} \right)^{3/2}} (\epsilon - E_c)^{1/2} \] (14)
Putting the above two equations together, the concentration of states is thus:

\[ n = \int_{E_c}^{\infty} D_e(\epsilon)F_e(\epsilon) d\epsilon = 2 \left( \frac{m_e kT}{2\pi\hbar^2} \right)^{3/2} e^{(\mu - E_c)/kT} \quad (15) \]

A similar exercise can be repeated for the density of the p-carriers, which results in:

\[ p = \int_{-\infty}^{E_v} D_e(\epsilon)F_e(\epsilon) d\epsilon = 2 \left( \frac{m_h kT}{2\pi\hbar^2} \right)^{3/2} e^{(E_v - \mu)/kT} \quad (16) \]

where \( m_h \) is the reduced mass of a hole in the valence band.

Equations (15) and (16) can be multiplied together to obtain an expression for \( np \) that eliminates \( \mu \) in favor of the band gap energy, \( E_g \):

\[ np = 4 \left( \frac{kT}{2\pi\hbar^2} \right)^{3/2} (m_e m_h)^{3/4} e^{-E_g/kT} \quad (17) \]

This result is known as the equilibrium relation, and it is important because the product of the electrons and the holes will always be constant, even if there are impurities introduced to the samples. However, if the semiconductor is intrinsic, i.e. no impurities have been added, then an excitation of an electron to the conduction band results in a hole in the valence band. We can use the fact that \( n = p \) to arrive at the final result, taking the square root of (17):

\[ n_i = p_i = 2 \left( \frac{kT}{2\pi\hbar^2} \right)^{3/2} (m_e m_h)^{3/4} e^{-E_g/2kT} \quad (18) \]

For a purely intrinsic semiconductor, the number of charge carriers, and thus the conductivity, goes as \( \sim e^{-E_g/2kT} \). This result will be used in the experimental section to plot the conductivity versus the temperature.
Extrinsic Conduction (Doping)

What happens when impurities are introduced to the sample? The conductivity of semiconductors can be dramatically increased by the addition of certain impurities, also known as doping. For example, the addition of boron to silicon in a ratio of as little as 1 in $10^5$ can increase the conductivity of silicon by as much as $10^3$ [Kittel]. Semiconductors such as silicon and germanium have four valence electrons and thus adopt a diamond structure, with each atom forming four covalent bonds with each of its nearest neighbors. When an impurity is added to the semiconductor, it will replace one of the atoms and form new bonds, as shown in figure 4.

If the impurity has valence five, such as arsenic, antimony, or phosphorous [Kittel], then after it has formed bonds with the nearest neighbors there will be one electron left over that is used for conduction. These types of impurities are known as donors because they “donate” an electron to be used for conduction.

However, these extra electrons are still initially bound to the impurity and must be thermally ionized before the conductive effects can be seen. This donor ionization energy can be determined by using a
modified version of the Bohr model of hydrogen, taking into account the dialectric constant and the effective mass of the atom:

\[ E_d = \frac{e^4 m_e}{2(4\pi\epsilon_0 \hbar)^2} = \left(\frac{13.6 m_e}{e^2 m}\right) eV \] (19)

If the impurity has only three valence electrons, it is known as an “accepter.” In order to replace a normal atom, the impurity has to borrow an electron from another covalent bond to complete its own bonds with its neighbors. This leaves a positively charged “hole” in one of the bonds, which like an excess electron, can be used to transport current. Since an acceptor is ionized when it accepts the extra electron, it requires an energy input similar to that of a donor. These impurities ionize much quicker than the intrinsic carriers and thus their effects can be seen more rapidly.

The prevalence of accepters or donors determines the type of semiconductor. If there are more accepters, then the conductivity is controlled by the positively charge holes, and the semiconductor is known as a \( p \) type. Similarly, if the donors are more prevalent, the conductivity is controlled by the negatively charged electrons, and is an \( n \) type semiconductor.

**The Hall Effect**

An important physical concept relevant to conductivity is the Hall Effect. The effect arises from the fact that a moving charge subject to a magnetic field will be subject to a force that is perpendicular to both the direction of travel and the magnetic field:

\[ \vec{F} = q \vec{v} \times \vec{B} \] (20)

This is shown in the figure below
The electrons in the sample are pushed upward, resulting in a net electric field inside the sample. This will produce a measureable potential difference between the top and bottom edges of the sample.

But suppose the charge carriers in the sample are holes instead of electrons. The figure above would still look the same since holes carry opposite charge, but also travel in the opposite direction. The only difference would be the direction of the electric field, which would result in a voltage opposite that of the electron case. The direction of the Hall voltage can be used to determine whether a semiconductor is p-type or n-type.

Additionally, if the Hall coefficient for a sample is known, it can be used to verify the density of the charge carriers. The Hall Effect is defined by the following formula:

\[ R_H = \frac{E}{jB} \] (21)
Where $E$ is the electric field produced between the top and bottom edges of the sample and $j$ is the current density. However, we know from Maxwell’s equations that

$$|E| = v|B| \quad (22)$$

Substituting the equation derived earlier, $v$ can be replaced with the current $I$:

$$E = \frac{IB}{nqA} \quad (23)$$

This gives an expression for the Hall Voltage

$$V_H = \frac{IB}{nqd} \quad (24)$$

Where $d$ is the depth of the sample. Formula can also be substituted back into formula to get an expression for the Hall coefficient

$$R_H = \frac{1}{nq} \quad (24)$$

The Hall coefficient is simply the inverse of the density of the charge carriers times the amount of charge each are carrying.

**Seebeck Effect**

The Seebeck coefficient is a thermoelectric effect that results from the application of a temperature gradient to a material. When a heat source and heat sink are applied to opposite ends of a material, a voltage will be induced across the sample in the same direction as the temperature gradient. The Seebeck coefficient is given by the rate of change of the voltage with respect to temperature:
\[ S = \frac{dV}{dT} \] (25)

However, it is usually incredibly difficult to measure the Seebeck coefficient of a single material, so two different materials are often used, the Seebeck coefficient is given by the difference of the absolute Seebeck coefficients of the individual materials:

\[ S_{AB} = S_A - S_B = \frac{dV_{AB}}{dT} \] (26)

This gives the number of volts generated per Kelvin applied.

Figure 6: The Seebeck Effect

The Peltier Effect

The Peltier effect is closely related to the Seebeck Effect, and is essentially the same phenomena in reverse. Whereas a temperature gradient is applied to produce the Seebeck effect, in this case a voltage is applied to create temperature gradient. As seen in the figure below, one of the junctions of
the two materials will be heated and the other will be cooled. This effect can be reversed simply by applying the current in the opposite direction. The equation describing the Peltier effect is given by:

\[
\frac{dQ}{dt} = I_{AB} I \quad (27)
\]

In short, the heat absorbed per second at each junction proportional to both the Peltier coefficient and the current applied to the circuit.

![Figure 7: the Peltier Effect](image)

**The Thompson Effect**

The Thompson effect is different from both the Seebeck and Peltier Effects, in that it occurs in a single semiconductor. It is caused by creating two different temperature gradients in the same material, as shown in the figure:
The bottom of the material, C, is heated, creating temperature gradients from A to C and B to C. This heat dissipation creates an emf opposite the current in the AC region and in the same direction as the current in the CB region, measurable by the difference in the resistances $R_1$ and $R_2$. The amount of heat dissipated between two sections at different temperatures is given by the following equation:

$$\frac{dQ}{dt} = I \int_{T_1}^{T_2} \sigma_a dT \quad (28)$$

Where $I$ is the applied current, $T_1$ and $T_2$ are the temperatures of the two points in question, and $\sigma_a$ is the Thompson coefficient. Note that this energy dissipated is in addition to the normal Joule heat dissipated in a resistor, given by

$$W = I^2 R \quad (30)$$
The Figure of Merit

Using the coefficients of the above thermoelectric effects, a dimensionless quantity can be determined that measures the efficiency of the material. This quantity is known as the figure of merit and is given by the letters ZT. The diagram below illustrates how this figure of merit can be determined. The setup consists of an n-type and a p-type semiconductor made of the same material and joined by a heated surface at one end and a cooled surface at the other. Thompson heating will be ignored in this derivation as it is negligible compared to the other effects.

![Diagram of the figure of merit](image)

Figure 9: The Figure of Merit

The efficiency of this system is given by the ratio of the useful power delivered to the resistor and the power that is drawn from the heat source and wasted

$$e = \frac{W_u}{W_w}$$  (31)
Due to the Peltier Effect, the applied current will cause heat to gather at the hot and cold joints of the semiconductor, where the power dissipated is given by

\[ W_h = VI \]  (32)

But from Equation (26) above, the voltage and the induced current can both be expressed in terms of the Seebeck coefficient, which is the same for both materials

\[ V = ST_h \]  (33)

\[ I = \frac{S(T_h - T_c)}{R + r} \]  (34)

where \( r \) is the internal resistance of the semiconductors. Combining these expressions gives

\[ W_h = S^2(T_h - T_c) \frac{1}{(R + r)} \]  (35)

For the rest of the derivation, the following substitution will be used

\[ x = \frac{R}{r} \]  (36)

In addition to the power dissipated at the joints, some power is spent transferring the heat to the cooled junction, and is given by

\[ W_c = K(T_h - T_c) \]  (37)

where \( K \) is the thermal conductivity of the material. Finally, the Joule heating of the samples themselves is taken into account. The power dissipated in the sample is

\[ W_i = \frac{I^2r}{2} \]  (38)
The factor of 2 enters the equation because only half the power is transferred to the $T_h$ junction while the other half is transferred to the $T_c$ junction.

The useful power delivered to the resistor is given by

$$W_u = I^2R = \frac{S^2(T_h - T_c)^2x}{r(x + 1)^2} \quad (39)$$

The efficiency for the circuit can then be represented as a combination of these terms

$$e = \frac{W_u}{W_h + W_c - W_i} = \frac{1}{T_h(x + 1)} \left( \frac{(T_h - T_c)x}{1 + \frac{K}{S^2}r(x + 1)} - \frac{(T_h - T_c)x}{2T_h(x + 1)} \right) \quad (40)$$

Within this expression there is only one quantity that is dependent solely on the properties of the semiconductor, defined by $z$. The area and length terms in $K$ and $r$ can also be eliminated and expressed by the dimensionless quantities $\kappa$ and $\sigma$

$$\frac{1}{z} = \frac{K}{S^2} = \frac{\kappa}{\sigma S^2} \quad (41)$$

By taking the derivative of equation (40) with respect to $x$, finding the extreme, and plugging it back into the equation, the final expression for the figure of merit is arrived at

$$ZT = \frac{S^2\sigma}{\kappa} \cdot \frac{T_h + T_c}{2} = \frac{S^2\sigma}{\kappa} T_{av} \quad (42)$$

The figure of merit is a useful quantity because it incorporates all of the thermoelectric effects but also reveals the complexity of an object’s properties. In order to maximize the figure of merit, one must increase the Seebeck coefficient and electrical conductivity while decreasing the thermal conductivity.
II. The Experiment

Experimental Setup

The experimental setup consists primarily of four components:

1. The Carriage
2. The Probe
3. The Oven
4. Other Electronics

The carriage consists of a simple ceramic cylinder on which the contact surfaces are mounted. The current is channeled through the opposing plates and the potential difference is measured between the parallel screws. There are two thermocouple probes which will also be used to measure the temperature of the sample. The larger holes are used to mount the carriage to the probe.

Figure 10: The Carriage
The probe is essentially an extension for the electrical leads and consists of a hollow tube attached to two vacuum-sealed valves in a “T” form. The carriage is fastened to the end of the tube, and the electrical leads are attached to the sample. The top of the probe has a serial port through which information can be exchanged with the voltmeter. The glass tube can be placed over the end of the probe to allow vacuum sealing.

The oven is the device in which the samples are heated. The probe is placed into the top opening of the oven and fastened into place. The electronics control the temperature of the oven, as well as apply the current, measure the voltage and temperature, and provide information to the computer.
Procedure

Prior to each test, the sample was cleaned and sanded to ensure that any oxidation that may have built up on the surface was removed. The sample was then carefully placed in the carriage and the two current leads were fastened tightly against the surface of the sample, sometimes using a thin sheet of graphite to ensure the best possible contact. The two voltage probes were also tightened enough to make a clear indentation on the surface of the sample. Once the sample is in place, the glass tube is placed over the end of the probe and is bolted down. The whole apparatus is placed in the oven; however, before the test is performed the oxygen in the tube must be removed to avoid any oxidation. Using a simple vacuum pump, the oxygen is removed and replaced with argon. The inert argon allows for a simulation of atmospheric pressure without any other compounding effects. Once all the necessary attachments are made, the Labview program is enabled and the measurements are then made and recorded automatically. After the resistance measurements are made the sample’s dimensions are measured, allowing the computation of the conductivity.

The Labview Program

In order to reduce the error in taking measurements and allow the lengthy tests to run without constant supervision, it was decided that a Labview program be developed. The Labview software proved to be immensely useful; it was easily programmable, compatible with the current lab equipment, and allowed for precise measurements of the conductivity. The program used in the tests was developed primarily by Steven Moses over the summer of 2009. For these particular tests, the program was set to measure the resistance for a given voltage as the sample was heated from 50 K to 500 K at 25 K intervals and to collect the same measurements as the sample is cooled back down to room temperature. The oven is heated at 2 K/min and cooled at 1K/min to ensure that the sample does not
contract too quickly and lose contact, although contact was usually lost before the sample reached room
temperature.

The Samples

The samples used in this study are known as skutterudites, which are exotic materials that have
the chemical formula ReTmPn12, where the “Re” term stands for a rare earth metal, “Tm” represents a
transition metal, and “Pn” is a pnictide. In this experiment four different samples of this form are
studied, with the formulas BaxYbyInzCo4Sb12. The figure below illustrates the structure of a skutterudite;
in this case the ytterbium would be substituted for the uranium, the cobalt for the iron, and the
antimony for the phosphorous.

Figure 12: The structure of a skutterudite
The barium and indium in the sample act as dopants, and the concentrations vary between the samples. Studies have been done on these types of materials, including measuring both the electrical conductivity and the figure of merit (Yang et al.), but the addition of the dopants may produce changes in the material’s behavior at higher temperatures as extrinsic carriers are added into the samples.

The other sample used is Sr0.2Co4Sb12, which forms the same structure as a skutterudites even though strontium is not a rare earth metal, rather an alkali earth metal. Unlike the other samples, this material has not been doped with indium or barium and will serve as a useful reference point when observing the conductivity over time.

Data and Analysis

Each of the five samples was tested using the procedures described above and the conductivity of each sample was plotted against the temperature in degrees Celsius. Since the strontium sample is very different from the other samples it was put on a separate graph, as seen below.
The conductivity of the sample dips a bit in the 200-300° range but begins to pick up again at about 400° degrees. This phenomenon can be explained through lattice vibrations. At room temperature all of the extrinsic carriers (the sample may still have some even though it has not been intentionally doped) have been ionized and the conductivity is at or near a local maximum. However, very few of the intrinsic carriers have been ionized at room temperature and thus their contribution to the overall conductivity is low. An approximation for the total mobility, which is related to the conductivity, can be given by:

\[
\frac{1}{\mu} = \frac{1}{\mu_{\text{lattice}}} + \frac{1}{\mu_{\text{impurities}}} \tag{43}
\]

As the temperature increases the vibrations of the lattice plains increase, but the concentration of carriers remains nearly constant since the extrinsic carriers are already saturated. Thus the overall mobility is lowered, leading to a small reduction in conductivity that reaches a local minimum around 300°. Still increasing the temperature, the intrinsic conductors are finally being ionized in significant quantities and this eventually overtakes the effect of the vibrating lattices, leading to an overall increase in conductivity. If the data had continued farther into higher temperatures the conductivity still should have continued to increase.

The four samples of the form $\text{Ba}_x\text{Yb}_y\text{In}_z\text{Co}_4\text{Sb}_{12}$ were compiled on one graph to facilitate comparison between them.
This is quite a strange result. The conductivities of the samples are approximately ordered by their doping concentrations, and the higher concentrations had higher conductivities, as expected. But why is there a sharp decrease in conductivity? This decrease is much sharper (close to 50% reduction from the local maximum to the local minimum) than the previous sample (≈25% reduction in the same area).

Some of the same phenomena occur in these samples. The extrinsic conductors will be ionized at room temperature and the lattice vibrations will be subject to the same rule as above. Perhaps for some reason the lattices in these samples are more sensitive to the vibrations. The conductivity appears to level at it approaches 500°, and it would be interesting to perform the test again, going to higher temperatures and investigating whether the conductivity begins to increase again. Since the intrinsic
carriers usually do not ionize in significant quantities until the samples reaches higher temperatures, perhaps the conductivity will begin to rise again if the sample is brought to appropriate temperatures.

**Conclusions**

The samples, especially the Yb-Co-Sb compounds, showed some odd behavior that should be investigated in future tests, perhaps by taking the sample to a higher temperature. The samples seem to be especially sensitive to lattice vibrations as the temperature increases, and the conductivity dips significantly. Future tests on these samples could give a better insight into the relationship between the lattice mobility and carrier mobility.

However, it is not just the conductivity that is important. The conductivity is only a piece of the puzzle that is the figure of merit. The conductivity data collected with these samples can be used to calculate the figure of merit for these materials, and further tests should be undertaken to determine the thermal conductivities and Seebeck coefficients for the samples so that the full picture can be drawn.

**Acknowledgments**

I would like to thank my research advisor, professor Ctirad Uher, for providing me with the opportunity to perform research for him and for giving me experience in the lab that will certainly assist me in my future endeavors. I would also like to thank my fellow undergraduate research partner, Steven Moses, for helping me learn the lab equipment and procedure and developing the Labview program. Lastly, I would also like to thank another undergraduate partner that worked with me during the 2008-2009 year, Derek Vermuelen, whose senior thesis assisted me in writing my own.
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